Recent progress in oxide phosphor thin-film electroluminescent devices

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Abstract

The present status and prospects for further development of thin-film electroluminescent (TFEL) devices using oxide phosphors are described. High-luminance oxide TFEL devices have been recently developed using a new combinatorial deposition technique featuring rf magnetron sputtering with a subdivided powder target. In addition, new flexible oxide TFEL devices have been fabricated on an oxide ceramic sheet and operated stably in air above 200°C.

1. Introduction

New inorganic phosphors have recently attracted great interest for many applications in flat panel emissive displays and lamps. In particular, many multicomponent oxides have been newly developed as phosphors in white light-emitting diode (LED) and thin-film electroluminescent (TFEL) device applications. However, optimizing the phosphor characteristics of multicomponent compound phosphors has proved markedly difficult. Recently, we proposed a way of resolving this problem: a new technique utilizing combinatorial deposition by rf magnetron sputtering (rf-MSP) with a subdivided powder target in order to optimize the chemical composition and impurity content in phosphors and, as a result, greatly improve luminescent characteristics [1-3]. Using this technique, the luminous efficiency in electroluminescent (EL) and photoluminescent (PL) emissions was effectively increased in a number of inorganic phosphor thin films. In addition to phosphor development, recent innovations in flat panel emissive displays and lamps have frequently required fabrication on a flexible substrate. In particular, flexible organic light-emitting diodes fabricated on a flexible plastic sheet are being actively developed for flat panel display and lamp applications. On the other hand, flexible flat panel EL lamps that feature an emitting layer composed of a ZnS:Cu phosphor powder dispersed in an organic dielectric binder and printed on a flexible plastic sheet are already in practical use [4]. In addition, a new flexible EL device fabricated by combining sphere-supported inorganic TFEL technology with a polypropylene-$BaTiO_3$ composite sheet has been recently reported [5-7]. A flexible inorganic TFEL device fabricated using a plastic substrate and a sulfide phosphor thin-film emitting layer postannealed with a pulsed laser also has been reported [8]. Nevertheless, high temperature operation is difficult to achieve for flexible flat panel light-emitting devices using flexible plastic substrates and/or organic materials. Recently, new flexible inorganic TFEL devices fabricated on a flexible metal foil or flexible ceramic sheet have been reported [9-11].

In this paper, the present status and prospects for further development of oxide TFEL devices are described. In particular, high-luminance oxide TFEL devices have been recently developed using a new deposition technique featuring rf-MSP with a subdivided powder target. In addition, new flexible oxide TFEL devices have been fabricated on a 50μm-thick-oxide ceramic sheet and operated stably in air above 200°C.

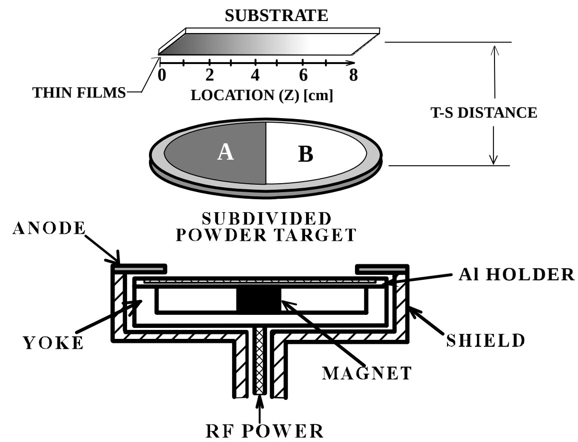

Fig.1 Schematic diagram of relationship between substrate and target subdivided into two parts.

2. Combinatorial Methods in New Phosphor Development

2.1 Combinatorial deposition by rf-MSP

The application of various combinatorial methods has proved useful in the development of new phosphor materials. Although most phosphor applications are used in either the thin-film or powder constituents, new developments have been recently reported using combinatorial thin-film deposition methods to deposit phosphor thin films with pulsed laser deposition (PLD) [12] and magnetron sputtering (MSP) in conjunction with multi-pellets or multi-targets as the source material. However, we have recently reported a new technique utilizing combinatorial deposition by rf-MSP with a subdivided powder target in order to enhance luminescent characteristics by optimizing the chemical composition and impurity content in phosphors [1-3]. By sputtering a powder target that is subdivided into two or more parts, phosphor thin films with a chemical composition and/or impurity content that varied across the substrate surface were successfully prepared. For example, phosphor thin films with a chemical composition or impurity content that varied across the substrate surface were produced by rf-MSP using a circular powder target composed of an Al holder divided into two sections, each containing different materials, as shown in Fig. 1. The different chemical compositions in part (A) and part (B) of the target could be either two different host materials or the same host material used with two different impurity contents.

As an example, the chemical composition (Ge content) and

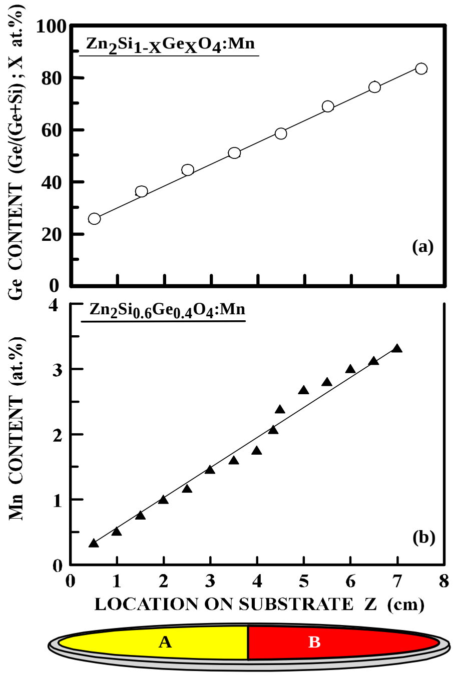

Fig.2 Ge content (a) and Mn content (b) as functions of location on substrate.

Mn content as functions of the location (z) on the substrate surface are shown in Figs. 2(a) and (b), respectively, for a $Zn_2Si_{1-X}Ge_XO_4$:Mn oxide phosphor thin film deposited on $BaTiO_3$ ceramic sheets and Si substrates (for evaluation of film thickness) by rf-MSP using a target subdivided into two sections, each containing a different powder [1,2]. In the combinatorial sputtering deposition that produced a variable Ge content, the two different powders, one of ZnO and SiO_2 with MnO dopant and the other of ZnO and GeO_2 with MnO dopant, were calcined in argon for 5 h at 900°C. In the combinatorial sputtering deposition with the variable Mn content, one powder was $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn with a Mn content of 0.2 at.% and the other was $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn with a Mn content of 3 at.%. The sputter deposition was carried out under the following conditions: atmosphere, pure Ar; pressure, 6 Pa; rf power, 140 W; and substrate temperature, 350°C. After deposition, the $Zn_2Si_{1-X}Ge_XO_4$:Mn phosphor thin films were postannealed for 1 h at 910°C. The thickness of all deposited phosphor thin films was approximately 1 μm. The Ge content (Ge/(Ge+Si) atomic ratio (X)) and the Mn content (Mn/(Mn+Zn+Si+Ge) atomic ratio) were measured by energy dispersive x-ray (EDX) spectroscopy and/or x-ray photoelectron spectroscopy (XPS). The host material of the postannealed phosphor thin films was identified as polycrystalline $Zn_2Si_{1-X}Ge_XO_4$ with a composition that depended on the location on the substrate surface. As can be seen in Figs. 2(a) and (b), the deposited $Zn_2Si_{1-X}Ge_XO_4$:Mn thin films featured a Ge content that increased monotonically from approximately 20 to 80 at.% across the substrate surface, and the deposited $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn thin films featured a Mn content that increased monotonically from approximately 0.3 to 3 at.%, respectively.

2.2 Phosphor developments in TFEL devices

The chemical composition and/or the impurity content in various multicomponent compound phosphors were optimized with the new combinatorial sputtering deposition in order to improve the EL characteristic in TFEL devices. The EL characteristics were investigated using a thick-ceramic-insulating-layer TFEL device structure [13]. The TFEL devices were fabricated by combining a phosphor thin-film emitting layer with a thick $BaTiO_3$ ceramic sheet (thickness of about 0.2 mm) insulating layer. The deposited phosphor thin film emitting layer with characteristics that varied across the substrate surface was first cut up into 5mm×5mm pieces and then postannealed. In the final TFEL device fabrication procedure, a transparent conducting Al-doped ZnO (AZO) thin film [14] and an Al thin film back electrode were deposited on the phosphor thin-film emitting layer and on the $BaTiO_3$ ceramic sheet, respectively. The PL and EL characteristics were evaluated using the cut up TFEL devices. The EL characteristics of the TFEL devices driven by an ac sinusoidal wave voltage were measured using a Sawyer-Tower circuit and a conventional luminance meter.

As an example, the chemical composition and Mn content of $Zn_2Si_{1-X}Ge_XO_4$:Mn phosphor thin films could be successfully optimized with the new combinatorial sputtering deposition, resulting in higher green emission intensities in EL and PL. Figure 3 shows typical data for obtained total PL emission intensity and obtained maximum luminance as functions of the Ge content of $Zn_2Si_{1-X}Ge_XO_4$:Mn phosphor thin films deposited, as above, on $BaTiO_3$ ceramic sheets, which were postannealed in an Ar gas atmosphere for 1 h at 910°C. The obtained maximum luminance shown in Fig. 3(b) was measured under an applied voltage at 1 kHz. As can be seen in Fig. 3(a), the intensity of green PL emission was strongly dependent on the Ge content; the spectral shape was relatively independent, whereas the wavelength at the peak

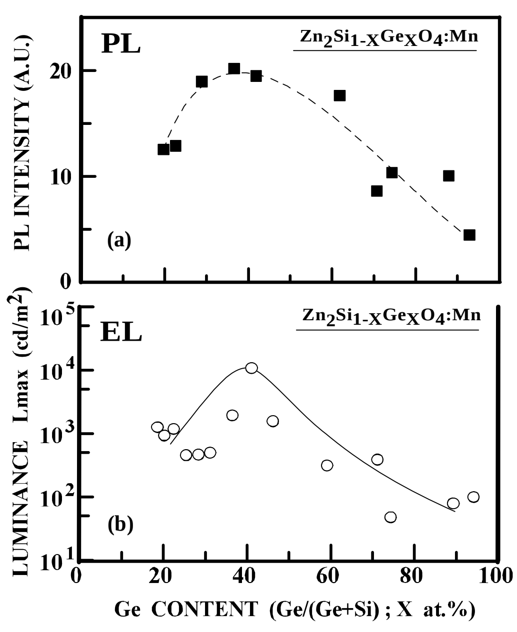

Fig.3 PL intensity (a) and maximum luminance (b) as functions of Ge content.

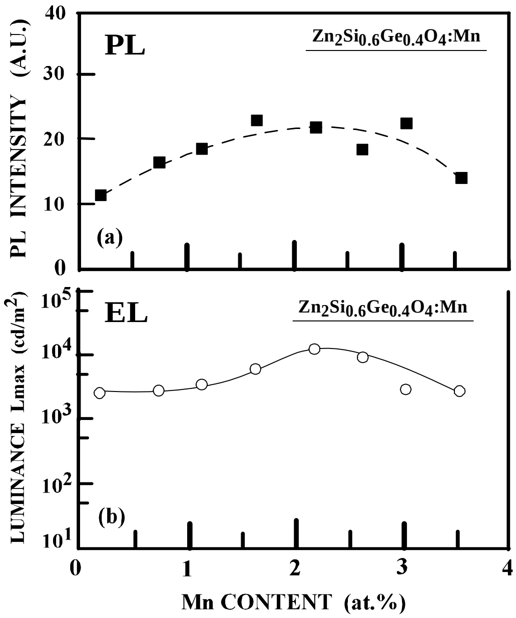

Fig.4 PL intensity and maximum luminance as functions of Mn content.

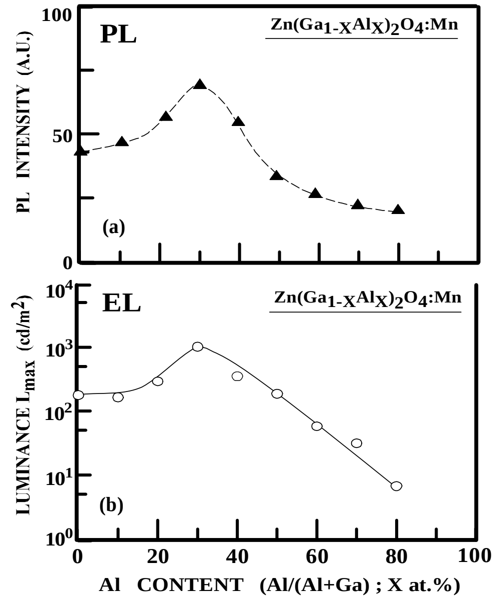

Fig.5 PL intensity (a) and maximum luminance (b) as functions of Al content.

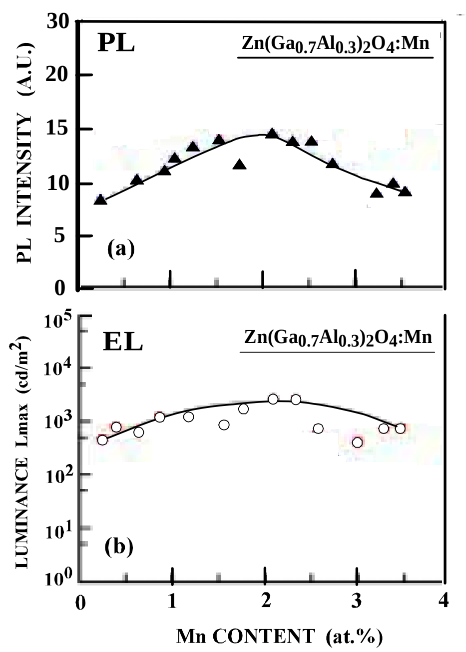

Fig.6 PL intensity and maximum luminance as functions of Mn content.

was slightly blue-shifted as the Ge content increased. It should be noted that both PL and EL exhibited the same Ge content dependence; the highest PL intensity and luminance were obtained at a location on the TFEL device corresponding to a $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn thin film, *i.e.*, a Ge content of approximately 40 at.%. These results show that the chemical composition (Ge content) of $Zn_2Si_{1-X}Ge_XO_4$:Mn multicomponent oxide phosphors could be optimized to obtain higher EL and PL emission intensities in only one deposition by using the new technique. Figure 4 shows obtained maximum luminance and PL emission intensity as functions of the Mn content of $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn phosphor thin films postannealed in an Ar gas atmosphere for 1 h at 910°C. The phosphor thin films were prepared, as above, on $BaTiO_3$ substrates with combinatorial sputtering deposition using a $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn powder target subdivided into two parts: one part doped with a Mn content of 0.2 and the other with 3 at.%. It should be noted that the highest luminance and PL intensity were obtained at a location indicating a Mn content of approximately 2.2 at.%.

TFEL devices fabricated using $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn thin films (Ge content of 40 at.%) with a Mn content of 2.2 at.% and driven at 1 kHz and 60 Hz produced high luminances of 11800 and 1536 cd/m^2 for green emission as well as high luminous efficiencies of 0.5 and 1.5 lm/W, respectively. Kitai et al. have reported that a high luminance of 479 cd/m^2 was obtained in a $Zn_2Si_{0.5}Ge_{0.5}O_4$:Mn TFEL device (Ge content of 50 at.%) driven at 60 Hz [15]. In addition, Minami et al. have reported a high luminance of 4220 cd/m^2 in a $Zn_2Si_{0.75}Ge_{0.25}O_4$:Mn TFEL device (Ge content of 25 at.%) driven at 1 kHz [16]. In comparison with these previously reported results, it can be concluded that this newly developed combinatorial sputtering deposition successfully optimizes the chemical composition and impurity content of $Zn_2Si_{1-X}Ge_XO_4$:Mn EL phosphor thin films, resulting in higher luminance for green emission.

As another example of oxide phosphors, the chemical composition (Al content, or atomic ratio (X)) and the Mn content of $Zn(Ga_{1-X}Al_X)_2O_4$:Mn multicomponent oxide phosphors were optimized with combinatorial sputtering depositions on $BaTiO_3$ ceramic sheets using a $Zn(Ga_{1-X}Al_X)_2O_4$:Mn powder target subdivided into two parts: one filled with $ZnGa_2O_4$:Mn powder and the other with $ZnAl_2O_4$:Mn powder. The Mn content (Mn/(Zn+Al+Ga+Mn) atomic ratio) of both powders was 2 at.%. The PL and EL characteristics were evaluated using $Zn(Ga_{1-X}Al_X)_2O_4$:Mn

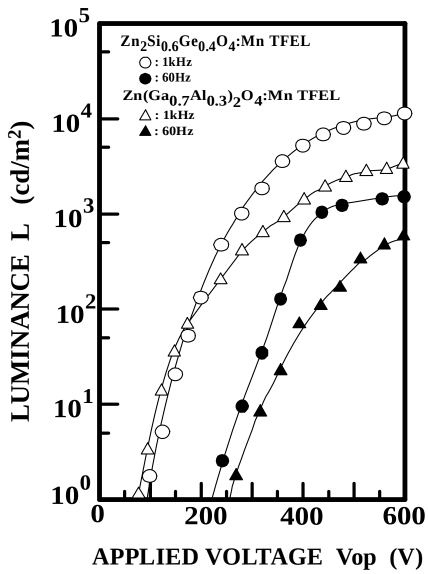

Fig.7 Typical L-V characteristics of TFEL devices with optimized oxide phosphor thin-film emitting layer.

thin films deposited under the following conditions: atmosphere, pure Ar; pressure, 6 Pa; rf power, 140 W; and substrate temperature, 350°C. The as-deposited thin films were postannealed in an Ar gas atmosphere for 1 h at 1000°C. Figure 5 shows typical data for obtained maximum luminance and obtained PL intensity as functions of the Al content for $Zn(Ga_{1-X}Al_X)_2O_4$:Mn TFEL devices: maximum luminance measured under an applied voltage at 1 kHz. It should be noted that both PL and EL exhibited approximately the same Al content dependence; the highest PL intensity and luminance were obtained at a location on the TFEL device corresponding to a $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn thin film, *i.e.*, a Al content of approximately 30 at.%. In order to optimize the Mn content in the $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn thin films, thin films were deposited, as above, on $BaTiO_3$ ceramic sheets using a $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn powder target subdivided into two parts: one part doped with a Mn content of 0.1 and the other with 3 at.%. Figure 6 shows obtained maximum luminance and PL emission intensity as functions of the Mn content of $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn phosphor thin films postannealed in an Ar gas atmosphere for 1 h at 1000°C. It was found that the highest luminance of 2200 cd/m^2 was obtained at a location indicating a Mn content of approximately 2.2 at.%. Minami et al. have also reported that a $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn TFEL device driven at 1 kHz produced the highest luminance, 1070 cd/m^2 [17]. Thus, it can be concluded that this newly developed combinatorial deposition technique successfully optimizes the chemical composition of $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn EL phosphor thin films, resulting in higher luminance for green emission.

Typical luminance versus applied voltage (L-V) characteristics are shown in Fig. 7 for TFEL devices fabricated using an optimized phosphor thin-film emitting layer, as described above, and driven by an ac sinusoidal wave voltage at 1 kHz: the highest luminance being 11800 cd/m^2 for green emission when using a $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn thin film prepared with a Mn content of 2.2 at.% and the highest luminance being 2200 cd/m^2 for green emission when using a $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn thin film prepared with a Mn content of 2.2 at.%. It can be concluded that this newly developed combinatorial sputtering deposition successfully optimizes the chemical composition and impurity content of various TFEL phosphors, resulting in higher luminance.

3. Flexible TFEL devices

3.1 TFEL devices using a flexible substrate

As mentioned above, Kitai's group has recently proposed a new flexible EL device fabricated by combining inorganic TFEL technology with a composite sheet composed of polypropylene and spherical phosphor film-coated $BaTiO_3$ particles [5-7]. They have demonstrated a flexible EL device fabricated with $Zn_2Si_{0.5}Ge_{0.5}O_4$:Mn oxide phosphor as the thin-film emitting layer by means of the following procedure: first using rf sputtering deposition to deposit a 700 nm thick $Zn_2Si_{0.5}Ge_{0.5}O_4$:Mn thin film on the top area of spherical $BaTiO_3$ particles with a size range of 53-63 µm and then annealing the phosphor-coated $BaTiO_3$ particles at 800°C. After forming a composite sheet composed of phosphor-coated $BaTiO_3$ spheres embeded into polypropylene, an ITO transparent electrode and a gold back electrode were prepared on the composite sheet by sputtering deposition. Under an applied voltage, the new highly flexible composite sheet EL device was green light-emitting and could be bent and rolled easily in any direction [7]. As another example of flexible TFEL devices fabricated using an organic substrate material, Yamazaki et al. have reported a flexible inorganic double-insulating-layer type TFEL device fabricated using a ZnS:Mn phosphor thin film deposited on a polyethylene sulfone (PES) substrate and then postannealed with a pulsed laser [8]. However, the obtained luminance from this flexible orange-emitting ZnS:Mn TFEL was relatively low. In contrast, Shepherd et al. have reported a flexible infrared emitting TFEL device fabricated without using any organic material [11]. A flexible double-insulating-layer type TFEL device was fabricated using a ZnS:ErF_3 phosphor thin film deposited on a Mo, W or steel foil substrate and then postannealed at 425°C in N_2 for 1 h. The green 550 nm EL emission as well as infrared EL emission could be observed from the ZnS:ErF_3 TFEL device with a Mo foil substrate flexed in tension and compression. In addition, we have recently proposed two types of flexible TFEL devices fabricated by forming a double-insulating-layer TFEL structure, as shown in Fig. 8, on a commercially available flexible zirconia-based ceramic sheet (Ceraflex, Japan Fine Ceramics Co., Ltd.) with a thickness of approximately 50-100 µm. Type (A) devices emit light through the top transparent electrode and the ceramic sheet substrate, while type (B) devices emit it through the ceramic sheet substrate. As a result, thin and flexible EL lamps using these two types of EL devices can provide different types of lighting.

3.2 TFEL devices using a flexible ceramic sheet

In TFEL device fabrication using a flexible ceramic sheet, a transparent conducting oxide (TCO) thin film was first deposited at 350-600°C as an electrode on the ceramic sheet substrate: for example, AZO and Ta-doped SnO_2 (TTO) thin films deposited by dc magnetron sputtering (dc-MSP) or rf-MSP [18,19] and Ga-doped ZnO (GZO) and indium-tin-oxide (ITO) thin films deposited by vacuum arc plasma evaporation (VAPE) [20]. It was found that both ITO and TTO thin films were stable in an Ar gas atmosphere at

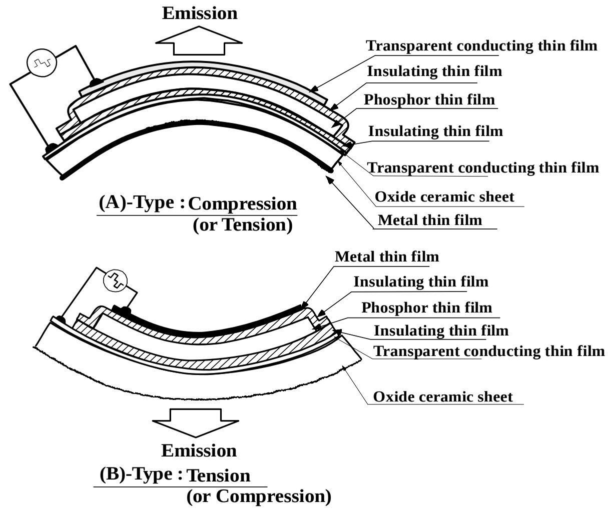

Fig.8 Schematic diagrams of two types of flexible TFEL devices.

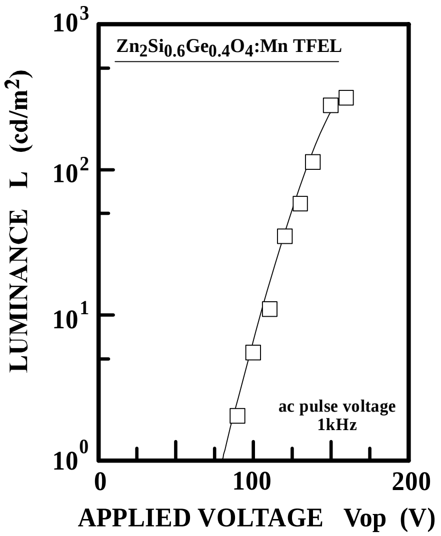

Fig.9 L-V characteristic of a $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn TFEL device driven at 1 kHz.

temperatures up to about 900°C. In particular, it should be noted that TTO thin films are more stable than other TCO thin films in an oxidizing atmosphere at temperatures above about 400°C [10]. The first insulating layer, composed of a BaO-Al_2O_3-Y_2O_3-TiO_2-Ta_2O_5 multicomponent oxide (BAYTO) thin film (thickness, approximately 200-500 nm) or a $BaTiO_3$ thin film, was formed on the previously deposited TCO electrode. The BAYTO film was deposited at 250°C by rf-MSP. A layered BAYTO/$BaTiO_3$ thin film was formed by then depositing a $BaTiO_3$ thin film (thickness, approximately 500 nm) with a sol-gel process, using a dip-coating with a commercially available solution (Kojundo Chemical Laboratory Co., Ltd.); a heat treatment in air was subsequently conducted at a temperature above 550°C. An oxide phosphor thin film was next deposited on the first insulating layer at approximately 300-350°C by rf-MSP. After this deposition, the oxide phosphor thin films were postannealed at 700-900°C in various atmospheres in order to

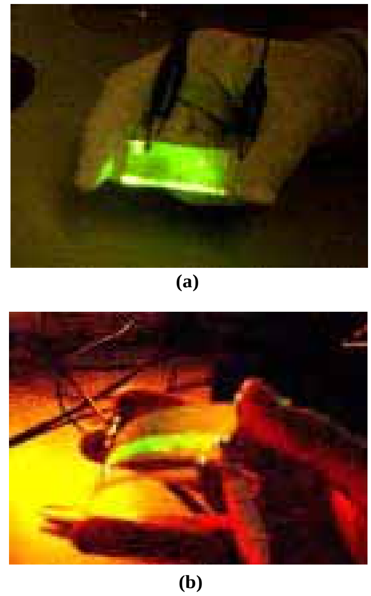

Fig.10 Photographs of light emission from a TFEL device: unstressed (a) and flexed in tension (b).

improve the luminescent characteristics of the phosphor thin-film emitting layer. Following this step, a BAYTO or Si_3N_4 thin film (thickness, approximately 200-500 nm) was deposited as the second insulating layer. The Si_3N_4 thin film was deposited at 250°C by rf-MSP. Subsequently, either a transparent conducting GZO thin film or an Al thin film was deposited as the transparent electrode in the type (A) device shown in Fig. 8 or as the back electrode in the type (B) device, respectively.

In inorganic TFEL devices, as described above, the process of postannealing the phosphor thin film emitting layers is very important in order to improve the obtainable EL characteristics, irrespective of phosphor type; the postannealing effect obtained is strongly dependent on the deposition method used and the condition of the phosphor thin films. It also has been reported that postannealing is essential for fabricating high-luminance TFEL devices using oxide phosphor thin films prepared by rf-MSP [21,22]. The use of a sintered ceramic sheet substrate in the newly developed flexible EL devices presented here has made it possible to perform heat treatments in various atmospheres at temperatures up to approximately 1000°C. When using oxide phosphors in TFEL devices, for example, suitable postannealing conditions for $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn thin films consist of an Ar gas atmosphere at about 900°C. As an example of an inorganic flexible TFEL device, Fig. 9 shows luminance as a function of applied voltage (L-V characteristic) for a type (A) TFEL device fabricated using a postannealed $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn thin film and driven by an ac pulsed voltage at 1 kHz. The device was fabricated by depositing a TTO, BAYTO, $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn, Si_3N_4 and GZO thin films as the TCO electrode, first insulating layer, emitting layer, second insulating layer and transparent electrode, respectively. The $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn thin film was postannealed in an Ar atmosphere for 30 min at 910°C. A high luminance of approximately 314 cd/m^2 for green

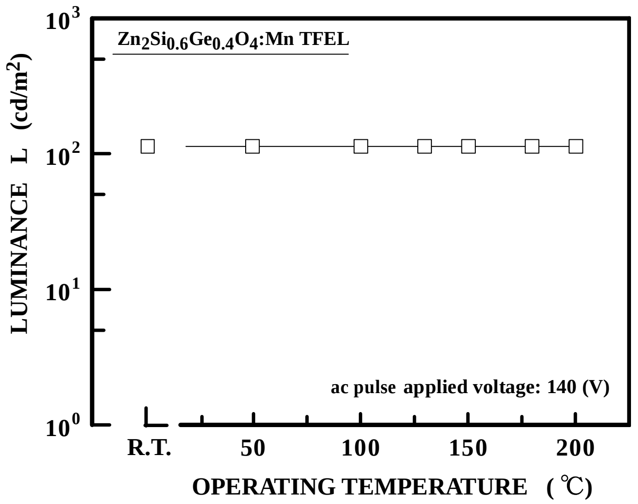

Fig.11 Operating temperature dependence of luminance from a $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn TFEL device driven with an applied voltage of 140 V.

emission was obtained in the TFEL device; Fig. 10 shows a photograph of light emission from a thin and flexible EL lamp constructed using a type (A) TFEL device fabricated with this emitting layer. It should be noted that in addition to light emission from the flexible $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn EL lamp, the device exhibited stable operation in air, even at a high temperature. Luminance as a function of operating temperature is shown in Fig. 11 for a $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn TFEL device driven by an applied voltage of 140 V at 1 kHz. This result demonstrates the stable operation of a flexible TFEL lamp using a $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn phosphor as the emitting layer in air at high temperatures above 200°C.

4. Conclusions

A new technique that incorporates a subdivided powder target to perform combinatorial deposition by rf magnetron sputtering has been proposed for the development of new phosphors. By sputtering with a powder target that is subdivided into two or more parts, phosphor thin films with a chemical composition and/or impurity content that varied across the substrate surface were effectively prepared. As a result of successfully optimizing the chemical composition as well as the Mn content in $Zn_2Si_{1-X}Ge_XO_4$:Mn and $Zn(Ga_{1-X}Al_X)_2O_4$:Mn multicomponent oxide thin-film phosphors using this technique, the luminance could be optimized in green-emitting $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn and $Zn(Ga_{0.7}Al_{0.3})_2O_4$:Mn thin-film electroluminescent (TFEL) devices. In addition, two newly developed types of flexible and thin inorganic TFEL lamps were fabricated by forming a double-insulating-layer TFEL device structure on a flexible oxide ceramic substrate (thickness of approximately 50-100 μm). New flexible $Zn_2Si_{0.6}Ge_{0.4}O_4$:Mn TFEL devices were fabricated on 50μm-thick-oxide ceramic sheets and operated stably in air above 200°C.

5. Acknowledgments

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